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IS 8979 (1997): Tetramethyl Thiuram Disulphide [PCD 13: Rubber and Rubber Products]

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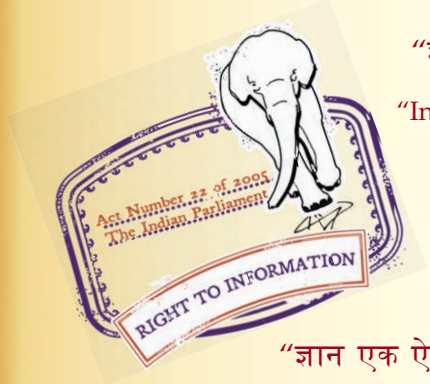

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(दूसरा पुनरीक्षण)

Indian Standard

TETRAMETHYL THIURAM DISULPHIDE — SPECIFICATION

(Second Revision)

ICS 83.040.20; 71.100.99

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

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Price Group 3

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Rubber Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Tetramethyl thiuram disulphide is an ultra-fast accelerator for vulcanization of rubber. When used alone with sulphur, specially in natural rubber base stock, it tends to be rather scorchy. It is more commonly used as a booster or other accelerators or in non-elemental sulphur cures (efficient Vulcanization Cures).

Tetramethyl thiuram disulphide is insoluble in water and slightly soluble in benzene, and alcohol. It is soluble in chloroform. Tetramethyl thiuram disulphide disperses in rubber without difficulty. Its solubility in rubber is approximately 0.125 percent by mass.

As per the present state of knowledge, Tetramethyl thiuram disulphide is approved by FDA (USA) for use in rubber articles intended for repeated contact with food provided total accelerator content does not exceed 1.5 percent by weight of the rubber product. However, this accelerator is harmful if swallowed and may cause skin irritation and sensitization. Whilst TMTD is not an acute eye irritant; prolonged exposure may cause development of chronic conjuctivitis.

This standard was first published in 1978. The first revision was done in 1986 with a view of include a more accurate method for determination of assay based on ultra violet spectroscopy and included the requirements and method for determination of particle size.

The Committee responsible for the preparation of this standard decided to update it in the light of experience gained. In this revision the assay of uncoated tetramethyl thiuram disulphide has been deleted and the requirements of manganese have been modified

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

TETRAMETHYL THIURAM DISULPHIDE — SPECIFICATION

(Second Revision)

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for tetramethyl thiuram disulphide (TMTD) intended for use as an accelerator in rubber compounds.

2 NORMATIVE REFERENCES

The following Indian Standards contain provision which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on the standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water — Specification (*third revision*)
1675 : 1971	Stearic acid, technical (*first revision*)
1683 : 1973	Barytes for rubber industry (*first revision*)
3399 : 1973	Zinc oxide for rubber industry (*second revision*)
3400	Methods of test for vulcanized rubbers:
(Part 1) : 1987	Tensile stress-strain properties (*second revision*)
(Part 2) : 1995	Hardness (*second revision*)
3660	Methods of test for natural rubber:
(Part 7) : 1988	Determination of Mooney viscosity (NR 8) (*second revision*)
(Part 51) : 1972	Determination of ash, total copper, manganese, rubber hydrocarbon, viscosity (Shearing disk viscometer), and mixing and vulcanizing of rubber in standard compound (*first revision*)
4588 : 1986	Rubber, raw, natural (*third revision*)
6918 : 1972	Mercaptobenzothiazole
7086 (Part 1) : 1973	Methods of sampling and test for rubber compounding ingredients : Part 1

3 REQUIREMENTS

3.1 Appearance

The material shall be in the form of a white to creamy white oil coated powder or pellets, free from visible impurities.

3.2 The material shall also comply with the requirements given in Table 1 in conjunction with Annex A, when tested according to the methods prescribed in col 4 and 5 of the Table 1.

3.3 Compounding Performance Requirement

The material when compounded and tested in accordance with Annex B shall have its performance comparable to that of the sample previously approved by the purchaser.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed as agreed to between the purchaser and the supplier.

4.2 Marking

4.2.1 The packages shall be securely closed and legibly marked to furnish the following information:

a) Name of the material;
b) Indication of the source of manufacture;
c) Net mass of the material;
d) Lot and batch number;
e) Month and year of manufacture; and
f) The words 'HAZARDOUS. AVOID SKIN CONTACT'.

4.2.2 *BIS Certification Marking*

The packages may also be marked with the Standard Mark.

4.2.3 The use of Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act,* 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for use of the Standard Mark may be granted to manufacturers or producers may be obtained from Bureau of Indian Standards.

5 SAMPLING AND CRITERIA FOR CONFORMITY

5.1 Sampling

For the purpose of ascertaining the conformity of tetramethyl thiuram disulphide in a consignment to this specification, sampling as prescribed in **15** of IS 7086 (Part 1) shall be followed.

5.2 Number of Tests

Tests for all characteristics shall be conducted on a composite sample.

5.3 Criteria for Conformity

The lot shall be considered as conforming to the specification if the composite sample satisfies each one of the requirements.

6 TEST METHODS

6.1 Test shall be carried out according to the methods prescribed in Annexes A and B and the Indian Standard as indicated in col 5 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Requirements of Tetramethyl Thiuram Disulphide (TMTD)
(*Clauses* 3.2 *and* 6.1)

Sl No.	Characteristic	Requirement	Methods of Test, Ref to	
			Annex of This Standard	Cl No. of IS 6918
(1)	(2)	(3)	(4)	(5)
i)	Assay (tetramethyl thiuram disulphide content), percent by mass, *Min*:		A	—
	a) Granules	95.0		
	b) Oil coated	96.0		
ii)	Free amine, percent by mass, *Max*	0.5	A	—
iii)	Melting point,°C *Min*[1)]	145	—	A-3
iv)	Residue on 150-micron IS Sieve, percent by mass, *Max* (in case of powders only)	0.5	—	A-4
v)	Loss on heating at 65°C for 2 h, percent by mass, *Max*[1)]	0.5	—	A-5[2)]
vi)	Sulphated ash, percent by mass, *Max*	0.5	—	A-6
vii)	Copper, ppm, *Max*	20	—	A-7
viii)	Manganese, ppm, *Max*	20	—	A-8

[1)]In case of dispute on melting point requirement, the final criteria for acceptance shall be the assay requirement.

[2)]In this case, the temperature of the oven is to be kept at 65±1°C instead of 105±2°C.

ANNEX A

[*Clauses* 3.2 *and* 6.1, and *Table* 1, *Sl No.* (i) *and* (ii)]

DETERMINATION OF ASSAY AND FREE AMINE CONTENT OF TETRAMETHYL THIURAM DISULPHIDE

A-0 GENERAL

Three methods, Method I, Method II and Method III are given. For routine work, the first two methods may be followed while for more accurate work, spectroscopic method given later should be followed which will also be the referee method in case of dispute. Free amines are determined by Method I only.

A-1 METHOD I

A-1.1 Principle

A solution of the substance is reduced with hydrogen sulphide and the liberated amine is titrated with acid.

A-1.2 Reagents

A-1.2.1 *Solvent Mixture* — isopropanol-toluene mixture in the ratio of 5:3.

A-1.2.2 *Standard Hydrochloric Acid Solution* — 0.1 N and 0.5 N.

A-1.2.3 *Sodium Hydroxide Solution* — 0.1 N.

A-1.2.4 *Bromophenol Blue Solution* — 1 percent solution in ethanol.

A-1.3 Apparatus

A-1.3.1 *Kipps Apparatus* — for hydrogen sulphide generation.

A-1.4 Procedure

Weigh accurately 1.5 g of substance into 500-ml conical flask. Add 150 ml of isopropanol-toluene mixture to dissolve the material, slightly warming the flask, if necessary. Titrate any free amine with 0.1 N hydrochloric acid using bromophenol blue indicator. Neutralize with a few drops of 0.1 N sodium hydroxide and pass hydrogen sulphide through the same for 15 minutes. Titrate the liberated amine with 0.5 N hydrochloric acid.

A-1.5 Calculations

A-1.5.1 Tetramethyl thiuram disulphide, percent by mass $= \frac{B \times 6}{M}$

A-1.5.2 Free amine, percent by mass $= \frac{A \times 45 \times 6}{M \times 1\,000}$

where

A = ml of 0.1 N hydrochloric acid required in the first titration;

B = ml of 0.5 N hydrochloric acid required in the second titration; and

M = mass of the sample in g taken for the test.

NOTE — 1 ml of 0.5 N Hydrochloric Acid = 60 mg of tetramethyl thiuram disulphide.

A-2 METHOD II

A-2.1 Principle

Tetramethyl thiuram disulphide on digestion with a dilute mineral acid undergoes decomposition and yields a salt of dimethylamine. After boiling off the volatile sulphur compounds, the amine is liberated by steam distillation under alkaline condition and estimated by titration with a standard acid.

A-2.2 Reagents

A-2.2.1 *Standard Hydrochloric Acid Solution* — 0.2 N.

A-2.2.2 *Boric Acid Solution*

20 g per litre. Each litre of solution shall also contain 10 ml of 0.05 percent methyl red and 0.7 ml of 0.35 percent methylene blue solutions.

A-2.2.3 *Sulphuric Acid Solution* — approximately 5 M.

A-2.2.4 *Sodium Hydroxide Solution* — approximately 5 M.

A-2.2.5 *Methyl Red Indicator Solution* — 0.5 percent.

A-2.3 Apparatus

A simple apparatus for steam distillation consists of two one-litre flat-bottomed flasks. One of the flasks used as 'steam generator' is provided with a long safety tube dipping well below the surface of water and a steam outlet tube. The other flask in which steam distillation is carried out is provided with a dropping funnel, a steam inlet tube which is connected to the steam outlet tube of the steam generator and a steam outlet tube which in its turn is connected to a water-cooled upright Liebig condenser through a splash head. The distillate from the condenser is collected in a 500-ml conical flask by dipping the bottom end of the Liebig condenser

below the liquid surface in the conical flask placed in a cold bath.

A-2.4 Procedure

Weigh accurately about 0.5 to 0.8 g of the prepared sample into a 150 ml round bottomed flask and add to it 50-ml of 5 M sulphuric acid. Attach a water-cooled reflux condenser to the flask and heat the contents slowly to boiling with occasional shaking. Allow to reflux for one hour, cool and transfer the contents quantitatively into the distillation flask of the steam distillation assembly. Steam distil until 200-ml of distillate is collected. Stop the distillation and reject the distillate which contains carbon disulphide, etc. Add a few drops for methyl red indicator into the contents of the distillation flask followed by 7.5 M sodium hydroxide solution until alkaline, add 10 to 15 ml in excess and steam distil the liberated amine. Collect about 250-ml of distillate in the receiver containing 50-ml of boric acid solution and titrate with 0.2 N hydrochloric acid to violet end point. Carry out a blank test on the reagents.

A-2.5 Calculation

Tetramethyl thiuram disulphide,

$$\text{percent by mass} = \frac{(T_2 - T_1)\, F \times 2.404}{M}$$

where

T_2 = ml of 0.2 N hydrochloric acid required for blank,

T_1 = ml of 0.2 N hydrochloric acid required for sample,

F = factor of 0.2 N hydrochloric acid, and

M = mass in g of sample taken.

A-3 METHOD III — SPECTROPHOTOMETRIC METHOD

A-3.1 Apparatus

Ultra Violet Spectrophotometer — Any standard instrument with the following provisions:

a) Range 150 nm to 500 nm,
b) Wavelength accuracy of not less than 0.2 nm,
c) Wavelength scan facility,
d) Recorder with speed control, and
e) Method UV cells (silica) — 1 cm path length — 2 pairs with teflon caps.

A-3.2 Reagents

A-3.2.1 *Isopropyl Alcohol*

A-3.2.2 *Methylene Dichloride*

NOTE — Both solvents should be of spectroscopic grade. In case such grade is not available, any grade for which the absorbance in a 1-cm UV cell (with air as reference) is less than 0.04 may be used.

A-3.2.3 0.8 percent solution of methylene dichloride in iso-propyl alcohol (*v/v*).

A-3.3 Procedure

A-3.3.1 Prepare a representative sample by taking about equal amounts from each sample container and mixing thoroughly. If the material is pelleted, crush the pellets before mixing. Weigh accurately 50 ± 3 mg of sample into a clean, dry, tared weighing funnel. Transfer the sample into a 100-ml volumetric flask (which has been thoroughly cleaned and rinsed with iso-propyl alcohol) by washing the sample through a funnel into the flask with 10-ml methylene dichloride. Swirl the contents until all the sample has dissolved, then pour 80 ml of iso-propyl alcohol through the weighing funnel into the flask. Make the volume up to 100-ml mark with iso-propyl alcohol by means of a dropping pipette. Stopper the flask and shake the contents until the solution is well mixed.

A-3.3.2 Wash out a clean dry 10-ml pipette with this solution, rejecting the washings. Pipette 10-ml of fresh solution into a clean 100-ml volumetric flask which has been rinsed with iso-propyl alcohol, taking every care to pipette an accurate volume. Make the volume up to 100-ml mark with iso-propyl alcohol by means of a dropping pipette. Stopper the flask and shake the contents until the solution is well mixed.

A-3.3.3 Fill two cells with a 0.8 percent (*v/v*) solution of methylene dichloride in iso-propyl alcohol. Put them in the 'sample' and 'reference' cell holders of the instrument. By controlling the 'zero knob', ensure that the absorbance in the 200-350 nm region remains zero. A scan on the records will indicate the actual variation; the absorbance line should merge with the chart base line. In any case, it is essential that at the specified wavelength (λ) for measurement of actual sample (*see* subsequent sections), the absorbance should be absolutely zero. Once the zero setting is done, do not touch the knob.

NOTE — Before putting UV cells in the holders, always hold them on the ground surfaces and wipe the transparent surfaces clean dry with soft tissue paper.

A-3.3.4 Take out the cell from the 'sample' holder, throw the solvent, dry the cell, rinse 4-5 times with the test solution, fill it up, put the cap, wipe clean the outer surfaces, and place it back in the holder. Do the wavelength scan at the slowest convenient speed between 200-350 nm. Measure accurately the absorbance (A) between the peak maximum and base line at the specified wavelength (in this case 275 nm).

NOTE — The shape of the absorbance curve should be similar to that of a standard sample.

A-3.3.5 *Standard Sample and Pure Material* — In case the absorption coefficient (E) of the accelerator is unknown, prepare a pure sample by recrystallizing at least 5 times from a solution of it in a suitable solvent. Preserve the final recrystallized sample in a sealed tube or under nitrogen in a refrigerator. Determine the absorbance (Ao) following the procedure **A-3.3.3**.

A-3.4 Calculation

$$E = \frac{A}{Cl}$$

where

A = absorbance of accelerator solution,
C = concentration of the solution in g/litres, and
l = path length = 1 cm.

$$\text{Active ingredient (percent)} = \frac{100A}{CE} = \frac{100ACo}{AoC}$$

where Co is the concentration of pure accelerator solution.

ANNEX B

(*Clauses* 3.3 *and* 6.1)

METHOD FOR COMPOUNDING AND TESTING OF TETRAMETHYL THIURAM DISULPHIDE

B-1 TEST COMPOUND

B-1.1 As a guidance, the following test compound may be used for testing performance properties of tetramethyl thiuram disulphide in rubber compounds:

Material	*Part by Mass*
Natural rubber, grade IS NR:5 (*see* IS 4588)	100
Zinc oxide (*see* IS 3399)	5
Stearic acid (*see* IS 1675)	1
Barytes (*see* IS 1683)	75
Sulphur (rubber grade)	2
Tetramethyl thiuram disulphide	0.5

B-2 COMPOUNDING

B-2.1 Follow the procedure prescribed in IS 3660 (Part 51).

B-3 TESTS

B-3.1 The tests given below are recommended for each test sample. The approved sample shall also be tested side by side using the same master batch, that is, compound excluding accelerator only.

B-3.1.1 Mooney scorch test shall be done at 110°C in accordance with the method prescribed in NR:8 of IS 3660 (Part 7).

B-3.1.2 Tensile strength, modulus at 300 percent elongation and elongation at break at different cures (for example 5, 7.5, 10 and 12 minutes) at 141°C from below to above the expected optimum cure shall be tested in accordance with the method prescribed in IS 3400 (Part 1).

B-3.1.3 Hardness on optimum cure at 141°C shall be tested in accordance with the method prescribed in IS 3400 (Part 2).

B-4 RESULTS

B-4.1 The values obtained with the test sample shall not vary by more than ±20 percent for Mooney scorch and ±10 percent for all the other characteristics from those obtained with the standard sample.

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